

Docket No. H 50019 HST

Remarks

This case is a continuation of PCT/JP02/10750. This preliminary amendment conforms the text of this application more closely to that of the PCT parent document. No new matter is being added. Support for the amendments can be found at page 10, line 29 to page 16, line 20 of the English-language translation of the PCT parent document. The Commissioner is hereby authorized to charge any required fees to Deposit Account No. 01-1250. Please direct any comments or questions regarding this amendment to the undersigned.

Respectfully submitted,



Mary K. Cameron
(Reg. No. 34,789)
Attorney for Applicants
248 589 4672

Henkel Corporation
Patent Law Department
2200 Renaissance Boulevard, Suite 200
Gulph Mills, PA 19406

MKC/mlc
G:\Data\H50019pre.doc

Mark-up of Changes to the Specification

[0018.] The cleaning process used for the metal wire rod is desirably selected from the group comprising, preferably consisting of shot blasting, sand blasting, bending, anodic pickling, and cathodic pickling. The object of the cleaning mentioned above is to remove the oxidized scale that forms as the result of annealing, etc., as well as various contaminants (oils, etc.). This cleaning process will allow the subsequent lubricant film formation to proceed smoothly. The reduction in the wastewater processing load is especially desirable from the standpoint of the environmental problems of recent years. For this, so-called mechanical descaling, a method that produces no wastewater, namely, inline shot blasting or bending, is useful. Moreover, when an acid is used, anodic pickling or cathodic pickling utilizing electrolysis can be used to shorten the processing time. For anodic pickling, an acid such as sulfuric acid is used. The wire rod is used as the anode and a counterelectrode (platinum plate, etc.) is used as the cathode to carry out electrolysis for several seconds under an applied voltage of 2–50 V and a current density of 2–100 A/dm² to cause part of the metal substrate to ~~[melt]~~ dissolve for the simultaneous removal of scale. For cathodic pickling, an acid such as sulfuric acid is used. The wire rod is used as the cathode and a counterelectrode (platinum plate, etc.) is used as the anode to carry out electrolysis for several seconds under an applied voltage of 2–50 V and a current density of 2–100 A/dm²; the force of the hydrogen gas that evolves from the metal surface is utilized to remove the scale.

[0032.] Drawing Workability: The wire rod was subjected to drawing in 3 steps under the conditions described below. The flaws detected during the third drawing step and the drawing load (kgf) in the third step were evaluated. ~~[The heavier the load without flaws the better.]~~ No imperfections (flaws) and a low drawing load are preferred, since the load should be low for the intended reduction in wire diameter.

- a. 1st step: Ø 3.00 mm → Ø 2.76 mm
- b. 2nd step: Ø 2.76 mm → Ø 2.40 mm

c. 3rd step: \varnothing 2.40 mm \rightarrow \varnothing 2.17 mm

[0033.] Heading: The wire rod for bolt forming was subjected to skin pass rolling (to \varnothing 8.3 mm). [2,000] 200 bolts were then prepared continuously with the use of an existing header machine and former oil as shown in Figure 1. The presence or absence of flaws on the front part was determined by the naked eye and the number and extent of the flaws were examined.

[0039.] In Comparison Examples 1 and 2, in which the lubricant/inorganic salt ratio was out of the range of the present invention, the lubricity was poor and partial fouling with the die occurred at the time of drawing; and no heading was possible. In Comparison Example 3 without the cleaning process, the lubricity was insufficient and further processing was impossible. In Comparison Example 4 with the coating weight out of the range, the lubricity was also insufficient. In Comparison Examples 5-7[4-7], in which the conventional technique was used, the lubricity was good but problems regarding processing space and environmental protection were present.

In the Examples

Processing Liquid 2

Inorganic Salt:	zinc phosphate + sodium tetraborate (weight ratio 1:2)
Lubricant:	[zinc] sodium stearate + calcium stearate (weight ratio 1: 1)
Lubricant/Inorganic Salt Ratio:	0.5
Solid Matter Concentration:	5%

Processing Liquid 3

Inorganic Salt:	[sodium] potassium tetraborate
Lubricant:	microcrystalline wax
Lubricant/Inorganic Salt Ratio:	2.0
Solid Matter Concentration:	[8%-] 10%

Processing Liquid 4

Inorganic Salt: [~~calcium~~] potassium sulfate + [~~sodium~~] potassium tetraborate
[~~(weight ratio 1:1)~~] (weight ratio 1:2)

Lubricant: PTFE

Lubricant/Inorganic Salt Ratio: 0.3

Solid Matter Concentration: 15%

Processing Liquid 5

Inorganic Salt: [~~calcium~~] potassium silicate

Lubricant: calcium stearate + polyethylene wax (weight ratio 1:2)

Lubricant/Inorganic Salt Ratio: 1.5

Solid Matter Concentration: [~~20%~~] 15%

EXAMPLES 4-5

The aforementioned substrate (wire rod) was subjected to the processing steps ① through ③ described below, sequentially according to the number. The line speed for processing was 40 m/minute.

- ① Shot Blasting: spherical shot (\varnothing 0.5 mm), time 10 seconds, pressure 5 kgf/cm²
- ② Surface Processing: processing liquid 4 (Example 4) or processing liquid 5 (Example 5) mentioned above was used; 60°C, immersion 2 seconds [~~3-seconds~~]
- ③ Drying: 140°C, 15 seconds (hot air blowing)

COMPARISON EXAMPLE 5

The aforementioned substrate (substrate steel ①, ④) was subjected to the processing steps ① through ⑧ described below, sequentially according to the number.

① Alkaline Degreasing: commercial degreasing agent (registered trademark Fine Cleaner 4360, a product of Nihon Parkerizing Co., Ltd), concentration 20 g/L, temperature 60°C, immersion 10 minutes

② Water Rinsing: tap water, standard temperature, immersion 10 minutes. [~~5-minutes~~]

- ③ Acid Rinsing: 17% hydrochloric acid, temperature 30°C, immersion 10 minutes.
- ④ Water Rinsing: tap water, standard temperature, immersion 5 minutes.
- ⑤ Conversion Processing: commercial zinc phosphate conversion processing agent (registered trademark Palbond 3670 X, a product of Nihon Parkerizing Co. Ltd.) concentration 90 g/L, temperature 80°C, immersion 10 minutes.
- ⑥ Water Rinsing: tap water, standard temperature, immersion 5 minutes.
- ⑦ Soap Processing: commercial reactive soap lubricant (registered trademark Palube 235, a product of Nihon Parkerizing Co., Ltd.), concentration 70 g/L, 80°C, immersion 5 minutes.
- ⑧ Drying: 80°C, 20 minutes.

COMPARISON EXAMPLE 6

The aforementioned substrate ([~~substrate~~] stainless steel ②) was subjected to the processing steps ① through ⑧ described below, sequentially according to the number.

- ① Alkaline degreasing: commercial degreasing agent (registered trademark Fine Cleaner 4360, a product of Nihon Parkerizing Co., Ltd), concentration 20 g/L, temperature 60°C, immersion 10 minutes
- ② Water Rinsing: tap water, standard temperature, immersion 10 minutes. [~~5 minutes.~~]
- ③ Acid Rinsing: nitric acid — hydrofluoric acid (10% nitric acid — 5% hydrofluoric acid), standard temperature, immersion 10 minutes.
- ④ Water Rinsing: tap water, standard temperature, immersion 5 minutes.
- ⑤ Conversion Processing: commercial oxalate conversion processing agent (registered trademark Felbond A, a product of Nihon Parkerizing Co., Ltd.), No. 1 agent concentration 30 g/L and No. 2 agent concentration 15 g/L, temperature 95°C, immersion 5 minutes. [~~15 minutes.~~]
- ⑥ Water Rinsing: tap water, standard temperature, immersion 5 minutes.
- ⑦ Soap Processing: commercial reactive soap lubricant (registered trademark Palube 235, a product of Nihon Parkerizing Co., Ltd.), concentration 70 g/L, 80°C, immersion 5 minutes.

⑧ Drying: 80°C, 20 minutes.

COMPARISON EXAMPLE 7

The aforementioned substrate (substrate titanium ③) was subjected to the processing steps ① through ⑧ described below, sequentially according to the number.

① Alkaline Degreasing: commercial degreasing agent (registered trademark Fine Cleaner 315, a product of Nihon Parkerizing Co., Ltd), concentration 15 g/L, temperature 60°C, immersion 10 minutes

② Water Rinsing: tap water, standard temperature, immersion 10 minutes. [~~5 minutes.~~]

③ Acid Rinsing: nitric acid — hydrofluoric acid (10% [~~15%~~]nitric acid — 5% hydrofluoric acid), standard temperature, immersion 10 minutes.

④ Water Rinsing: tap water, standard temperature, immersion 5 minutes.

⑤ Conversion Processing: commercial conversion processing agent (registered trademark Palmet 3855, a product of Nihon Parkerizing Co., Ltd.), concentration 25 g/L, temperature 60°C, immersion 10 minutes.

⑥ Water Rinsing: tap water, standard temperature, immersion 5 minutes.

⑦ Soap Processing: commercial molybdenum system lubricant (registered trademark Palube 4649C, a product of Nihon Parkerizing Co., Ltd.), concentration 800 g/L, 80°C, immersion 3 minutes.

⑧ Drying: 80°C, 20 minutes.